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THE PHYSICS OF TABLET COMPACTION REVISITED

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This overview followed the chronology of the compaction event for powders in the die. It started with filling the powder into the die cavity, which is confined in the bottom by a punch tip; it then followed the events that occur as a result of the entrance of an upper punch into the die and the progression of the upper punch to close proximity to the lower punch, and the forces and responses to forces involved. The mechanism of compaction is discussed and is focused primarily on the work provided by David Train (1956)[1]. The effect of the force on the materials being compacted are discussed. The characteristics of the material being compacted are reviewed with regard to the specific properties of the material as it would relate to their compaction characteristics, and second, the nature and distribution of the force that was applied to the powder, and its role in the compaction event is discussed. With regard to material properties, the deformation characteristics of the powders are emphasized as well as the physical properties such as particle size, particle shape, surface properties of the particle, and crystallinity. The types of deformation properties and their

specific roles in the compaction event are discussed. The nature of the forces that exist between particles in the compact is discussed. The fact that dipole type forces, London dispersion forces and hydrogen bonding all have possible roles in the cohesiveness or adhesiveness of the material being compacted is presented. Typical pharmaceutical materials and their compaction characteristics are discussed from the standpoint of ascertaining their properties, relating them to their compaction, and evaluating the results of that compaction. Ejection force following compaction is discussed and the role of that event in the stress patterns exhibited by compacts is described. Machine characteristics and the effect of the tabletting machine design and capability on the compact formation are reviewed.

For the purposes of this article, this overview will be started by assuming that the powders have been filled into a die cavity and are confined on the bottom by a punch tip, and are awaiting the entrance of an upper punch into the die cavity to initiate the compaction process. Let us now examine what is happening to the individual powder particles as we progressively reduce the volume of the powder bed to the point of maximum compaction and closest proximity of the two punch tips. At the outset, after the powder is filled into the die cavity (Figure 1), and prior to the entrance of the upper punch into the die cavity, the only forces that exist between the particles are those that are related to the packing characteristics of the particles, the density of the particles, and the total mass of material that is filled into the die. The packing characteristics will be determined by the characteristics of the individual particles. Particle shape will have a major role in the packing. It is obvious that symmetrical, cubical particles will pack dramatically

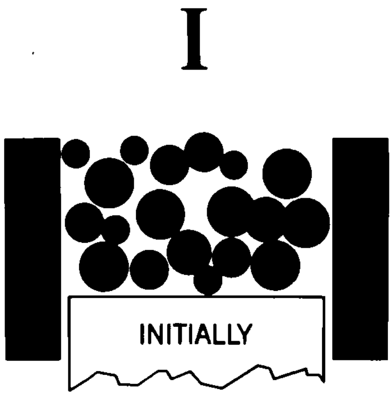

FIGURE 1

differently than filamentous, needle-like particles. The packing characteristics will also be a function of the blend of materials that are present. The packing characteristics of one material, which may be inherently very good, or approximate that of the cubical circumstance (sodium chloride) may well be offset by the shape factors introduced by a second excipient or active. in addition to particle shape, the surface characteristics of the particle can obviously affect the packing. The packing characteristics are important because they will define the amount of contact there is between particles. In the absence of compression force, there are several types of rearrangement of particles identified at low consolidation pressures.[1] The extremes types, the extremes of these were rearrangements such as simple particulate repacking, and to localized melting of asperities under high local stresses. Sodium chloride was studied at low compaction pressures (Hersey and Rees, 1970)[2] and again by Hersey, Rees and Cole (1973)[3] and they concluded that rearrangement of particles at low applied compaction pressures will depend upon the ratio of the particle size to the die cavity diameter and

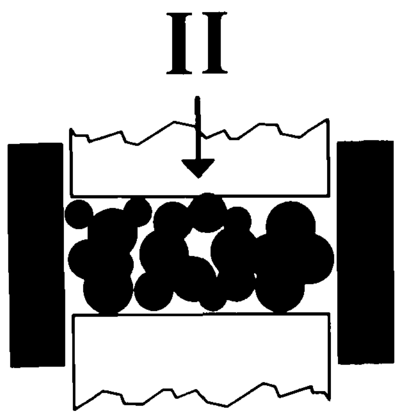

FIGURE 2

also to the packing configuration adopted by the powder as it is introduced into the die. For example, particles of a powder with poor flow characteristics may protect relatively large voids by bridging, and thus would be expected to undergo considerable rearrangement at low compaction pressures.

Having filled the die cavity with the powder blend, the next step in the compaction process is for the upper punch to enter into the die cavity and for the upper and lower punch tips to move ever closer in proximity (Figure 2), thus exerting ever greater applied pressure to the powder mixture. The powder compaction process was fairly well defined between the period of 1947 to 1962. The initial workers (Seelig and Wulff, 1947)[4] described what they envisioned as a three-stage operation. The initial stage was a packing rearrangement wherein energy losses to the applied energy are sustained through interparticulate friction. This stage is followed by elastic and plastic deformation, and here the losses

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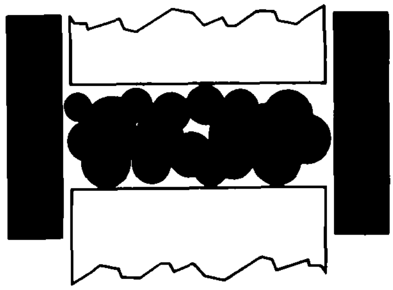

FIGURE 3

in energy are due to interparticulate and die wall friction, as well as deformation of particles. The third phase (Figure 3) was envisioned as "cold working." High residual stresses are set up leading to fracture of the structure. This work was followed in 1956 by Train[1] and his thesis provided a very meaningful study of the compaction process which he divided into four stages.

(1) A decrease in the relative volume is caused by interparticulate slippage of powder, leading to closer packing.

(2) When the particles are immobilized they are in the second stage. Here a formation of temporary structure (struts, vaults, and columns) takes place, which in turn protects small void spaces.

(3) Crushing or plastic flow characterize the material failure at this stage. In this stage the breakdown of the particle is taking place at point contacts. As these points are broken, in a confined space, new surfaces are produced and a bonding or cold welding[4] might take place. The coherent or adherent

properties of the material determine whether this does or does not take place.

(4) When the structure formed is strong enough to support the applied load, any further reduction in the volume of the compact involves the normal compressibility of the material being compacted.

If we accept Train's mechanism of compaction, the factors affecting the production of a compact might be divided into three main groups, namely: the physical properties of the powder; its flow and packing characteristics in the bulk condition; and its behavior under pressure. This work was followed in 1962 by Huffine.[5] He utilized sodium chloride, sucrose, and quartz in a study of the particle size effects in the compaction of powders; and he also postulated a mechanism of powder compaction. He stated that the decrease in volume as a result of applying pressure to a powder mass may take place in several ways: (1) particles may slide over each other without appreciable deformation to a new equilibrium position; (2) particles may elastically deform at or around the point of contact between particles; (3) particles may deform plastically; (4) the particles may deform by fracture and breakage. The characteristics of the material will dictate which mechanism will predominate. It is likely in most cases that a combination of two or more of the above possibilities will occur either simultaneously or consecutively as a function of the applied load.

Initially in a powder bed, there is essentially only point contact between particles. Application of an external force to the bed results in

FORCE FACTORS AFFECTING STRESS WITHIN COMPACTED POWDER BED

- MAGNITUDE OF FORCE
- RATE OF APPLICATION OF FORCE
- CONTACT TIME OF APPLIED FORCE
- PHYSICAL PROPERTIES OF THE MATERIALS

FIGURE 4

forces being transmitted through these interparticulate points of contact. The result of the force is that stress (Figure 4) will be developed at these points of contact and local deformation of the material follows. This deformation will be either elastic, plastic or destructive, depending upon (1) the rate of application of the external force, (2) the magnitude of the force, and (3) the duration of the locally induced stress as well as the physical properties of the material. So that there is a clear understanding of these deformation characteristics, let's define them as follows (Figure 5). Elastic deformation is when the change of shape of a stressed body recovers when the stress is released. Plastic deformation is defined as when the change of shape of a stress body does not recover when the stress is released. One reaches the elastic limit of a material when the stress at which the material deviates from linear elastic behavior is exceeded. Destructive deformation is described as deformation which results in fracture. The material is stressed at a magnitude or at a rate which exceeds the material's ability to withstand by either elastic or plastic deformation and responds by fracture.

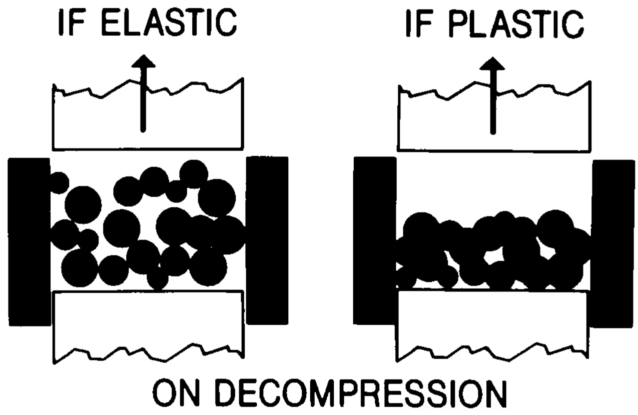

FIGURE 5

With regard to the compacting load, in the process of compaction the main factor affecting compaction is the compacting load. The magnitude of the compacting load, the rate at which the load is applied, the duration of the application of the load, and the characteristic transmission of forces throughout the compact to which the load is being applied are the primary points of consideration. The effect of the magnitude of the compacting load has been studied extensively by numerous investigators. The magnitude of the compacting load is the main factor determining the finished compact characteristics when measured in the usual pharmaceutical manner utilizing devices termed "hardness testers." This relationship between the applied force or load and the hardness of the finished compact implies that the cohesiveness of the particles within the tablet, or the bond strength created from particle to particle is related to the applied load. The primary function of the compacting load is one of increasing the true area of contact between particles, thereby increasing the strength of the bond between

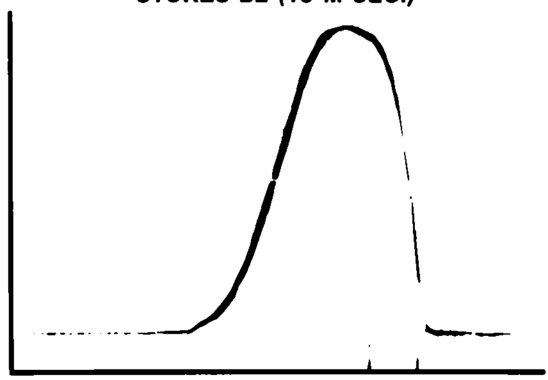

FIGURE 6

particles. The duration and rate of application of the compacting load are also major factors governing the effect of the compacting load. Application of a compacting load to material in a die imparts work to the material in the die. The amount of work applied to the material is a function of the time over which the load is applied. This is the "contact" time or duration of the compacting load (Figure 6). The greater the work done on the material being compacted, that is to say, the greater the area under the curve formed by plotting compacting load vs. time; the greater the true area of contact, and therefore potentially the greater the degree of cohesion or adhesion. The area under the curve can be increased by either increasing the magnitude of the load, or increasing the time to which the material is subjected to the load. The other factor with regard to compacting load is the rate of application of the load. This is a factor primarily because of the material's reaction to the load while being compacted. During the

SOME PHYSICAL PROPERTIES OF MATERIALS THAT AFFECT COMPACTION CHARACTERISTICS

- PARTICLE SIZE
- PARTICLE SHAPE
- PARTICLE CRYSTALLINITY
- SURFACE PROPERTIES OF PARTICLES
- DEFORMATION CHARACTERISTICS

FIGURE 7

second and third stages of the compaction process, where the particles are immobilized and are either being crushed or exhibiting their characteristic flow properties, the rate of application of load is a factor. If the material being compacted is incapable of deforming plastically at a rate sufficient to accommodate the applied load, the consequence will be that the material will fracture. This phenomenon is especially found in materials which are crystals of inorganic salts in which there are natural flaws and in which the increased strain due to stress causes failure in accordance with the griffith crack theory. Most materials can be caused to fail by applying sufficiently large loads, or by applying loads rapidly enough to overcome the critical deforming rate.

Other physical properties of the material affect the compaction characteristics (Figure 7). Crystallinity, particle size, particle shape, surface properties of the material, as well as the material's deformation

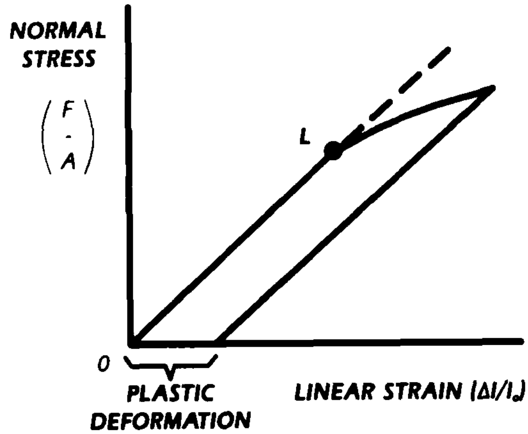

FIGURE 8

characteristics, discussed previously, are factors. A crystalline material will generally exhibit elastic deformation characteristics, while amorphous materials tend to be plastic deformers. Materials which elastically deform will exhibit compaction characteristics that are dependent upon this property. For example (Figure 8), if an elastically deforming material is compacted under a load which does not exceed the elastic limit of the material, or if the rate of application of the load does not exceed a value to which the material can elastically deform without fracturing, the material will show definite rebound tendencies. On the other hand, if the elastic limit is exceeded, or if the rate of application of the load is sufficiently rapid such that the material is incapable of deforming at a rate sufficient to accommodate the induced stress, the material will then fracture. If fracture occurs, then the rebound tendencies following compaction will be lessened. Amorphous materials tend to exhibit plastic deformation rather than elastic, and will compact accordingly. Generally, materials exhibiting plastic deformation will not exhibit the degree of elastic recovery on release of the compacting load. However, the critical relationship would probably be the ratio of the amount of elastic recovery to the amount of plastic deformation.

Particle size does affect compaction characteristics. In the work of Shotton and Ganderton (1961)[6], the effect of particle size on the bonding of materials was examined as the materials were compressed into tablets. They utilized sodium chloride, aspirin, and hexamine, and the results show that tablet hardness or force required to crush the tablets is a function of the particle size of the materials being compacted. This article also noted some very interesting observations that are worth discussion. At high pressures, the hexamine tablets tended to cap and laminate, and this effect increased progressively with increase in applied force. The results indicated that the production of a hexamine tablet of acceptable strength could be achieved by both reducing the particle size and the applied pressure. They attempted to determine the reason for capping, and showed that the tablets prepared under normal or evacuated conditions showed identical strength and capping characteristics, and therefore, assumed that entrapped air was not responsible for capping in this instance. They also further reported that capping was eliminated by coating the hexamine with stearic acid. Coating the hexamine does not markedly change the geometry of the deforming system, and, if entrapped air was the cause of capping, the capping still should occur. The stearic acid functioned to eliminate the capping by reducing the stress pattern produced by the ejection forces after the compaction. It was also reported in this publication[6] that particle size has an effect on the ejection forces. As particle size decreased, ejection forces were shown to increase. This occurs because the force lost to the die wall increases. That is to say, the quantity of force applied minus the force transmitted, increases. Ejection forces are derived from the shear of the area in proximity to the die wall, and this increase in ejection force as a function of particle size may be due to two things: (1) the increase in total contact area; or (2) the increase in the effective shear strength.

Particle shape affects compaction characteristics in that it affects packing characteristics. In the initial stages of the compaction process, there is a tendency for particle rearrangement to occur. If particle shape is such that rearrangement can occur readily, that is, if the particles are rather spherical in shape, then the compaction properties will be favorably affected. However, if the particle shape is irregular, and the particles do not pack easily, then we enter the second stage of compaction, which is the stage where the particles are fixed in position and the force is increased and begins to crush the temporary structure that has been formed. We will have more temporary structure formed in materials which exhibit poor packing characteristics, and the applied force required to crush those temporary structures will be greater than if the structures did not exist.

The surface properties of the material being compacted are also very important to the compaction characteristics of the material. Surface purity, crystalline perfection, total surface area and ionic character of the surface are a few of the properties that will influence the compaction characteristics of a material. Materials which are chemically pure and which have pure, uncontaminated crystalline surfaces, have the potential of cohering with a strength equal to the bulk strength of the substrate. Contamination by adsorbed moisture or gases, oxide layers, grease and foreign materials such as lubricants, reduce this cohesion, and it is in this contaminated state that the majority of the powders that we work with exist. These contaminated films must be penetrated during compaction so that clean areas come into contact and a cohesive bond is produced.

Now bonding will be addressed, as it takes place inside a compact, and the nature of the forces that exist between the particles in that compact will be discussed. Current theory in adhesive technology implies that molecular forces probably account for the majority of the strength of adhesive bonds. The forces existing between fine particles are too small to permit direct experimental determination. They must therefore be deduced from observations on larger bodies, on powder beds, and from the properties of aggregates and dispersions. Many of the properties of a bulk powder, especially its flow properties, are influenced by interparticulate cohesion or by the adhesion of the particles to surfaces. The measurement of such properties is an indication, although it is an indirect, relative one, of particle stickiness. The most direct and most easily understood test for the measurement of the cohesiveness of a powder is a measurement of the force required to split a powder bed (Figure 9). Correlations have been reported between the tensile strength of a powder bed and its fineness, degree of compaction or compacting load, and the presence or absence of shear forces on the powder bed. A paper by Gregory (1962)[7] discussed the effects of applying shear (Figure 11) as well as compaction pressure on the briquetting of a coal

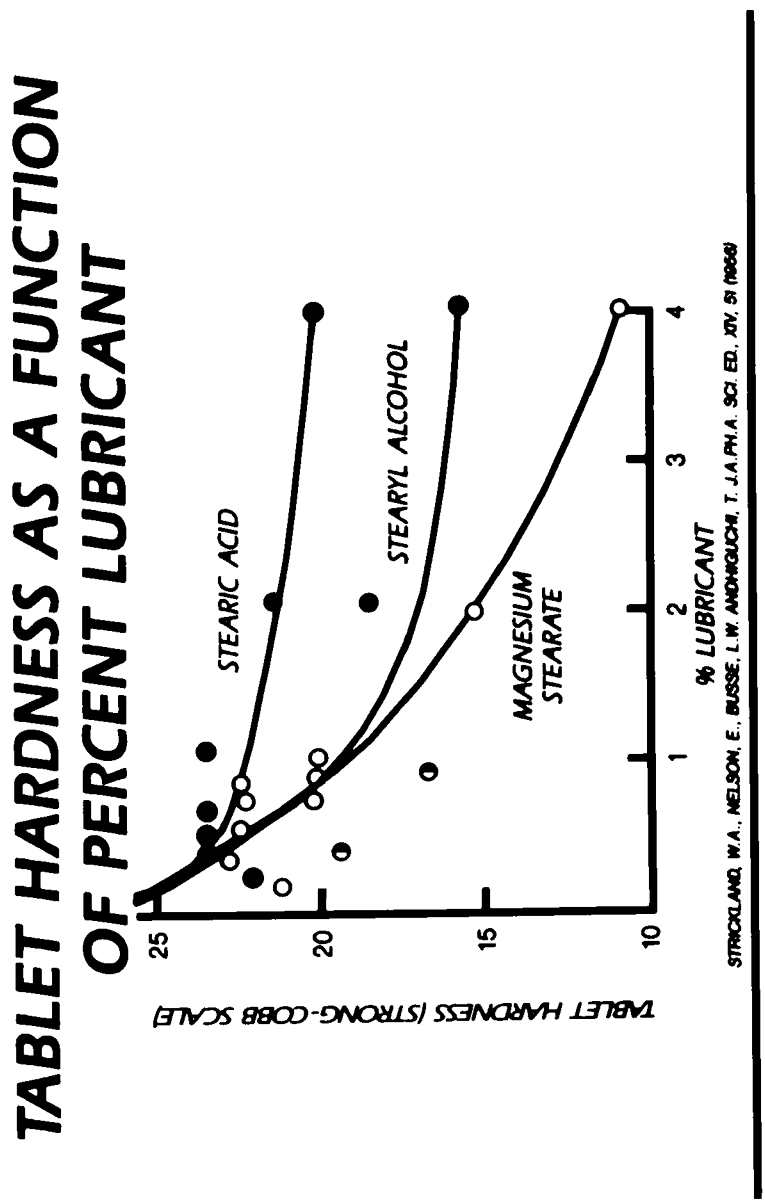
TABLET HARDNESS AS A FUNCTION OF PERCENT LUBRICANT
TABLET HARDNESS (STRONG-COBB SCALE)
25
20
15
10
STEARIC ACID
STEARYL ALCOHOL
MAGNESIUM STEARATE
1
2
3
4
% LUBRICANT
STRICKLAND, W.A., NELSON, E., BUSSE, L.W. ANDHIGUCHI, T. J.A.PH.A. SCI. ED. XIV, 51 (1956)

FIGURE 11

powder. Coal, considered a semi-plastic material, exhibits compact strength, for any given size distribution at any given mode of preparation, related to the density of the compact. To obtain compacts of high strength, it is necessary to insure that the compaction technique is effective in bringing about the necessary amount of increase in density, and that the compact suffers no damage during ejection from the die. The density and strength are determined by the pressure that is applied, but ultimately they approach limiting values which are not increased by further increase in pressure. This maximum density of the compact falls short of the density of the powder by an appreciable percent. This failure to achieve complete densification arises from two causes. First, as the pressure is being applied, it is opposed by forces set up in the powder and also by frictional forces on the walls of the die which resist the movement of particles that prevent intimate contact between the surfaces of the particles. Second, when the external pressure or compaction force is removed, the deformed particles recover their shape partially, and the compact expands. As a result of this expansion, the voids within the tablet increase and residual strains remain. A large elastic recovery is associated with a weak compact. Gregory[7] reported that it was possible to materially increase the density of a powder compact at a given compression force if, while under compression force, it is subjected to a shear stress. The application of this shear stress increases the compact strength and reduces the elastic recovery. The gain in strength with the application of additional shear stress under load may be substantial, but the full benefit is obtained only if the shear strain is produced under maximum compression force. In the case of coal, as reported by Gregory,[7] shear strain introduced at pressures below two thirds the maximum was of no value.

In pharmaceutical tablet compression, it is generally accepted that the cohesive nature of the material with which we are working is inadequate to achieve the degree of cohesion we need to have to make an adequate tablet. We therefore utilize processes or additives which improve the compaction characteristics of the material with which we are working. Binders or adhesives are added to increase the adhesiveness of the materials with which we are working. The theory of the use of an adhesive or binder depends upon the cementing of materials by some product which is strongly adsorbed by each surface, and so as to bond the surfaces together. An adhesive may be added in the form of a dry powder or liquid. It must possess cohesive strength besides being able to exert strong adhesion to the materials that you wish to bond. Adhesion is believed to result from the same forces between molecules that are responsible for the physical properties of a pure substance. The problem of using an adhesive is the problem of utilizing these forces at an interface. A solid is held together by fields of force around each ion, atom or molecule. At the surface of the solid, these fields of force do not terminate, but continue into the space beyond. They can attract molecules of a liquid as in wetting, or they can attract molecules, atoms or ions of another solid as in adsorption or adhesion. In actual fact, mutual attraction occurs since the surfaces of particles of the attracted substance, such as an adhesive, also possess fields of force. This attraction is approximately proportional to the fields of force, and it should be possible to calculate the field of force adjacent to the surface of a solid, and thus predict quantitatively the attraction of the solid for another body. In practice, precise calculations of this nature are not possible. Among the more important forces responsible for the field of force adjacent to the surface of solids are London's dispersion forces, electrostatic forces, and hydrogen bonding. London's dispersion forces are intermolecular forces which act between all atoms, regardless of polarity or electrical charge. They owe their origin to the fluctuating electrical moment produced by movement of the electrons in their atomic orbits. They are capable of inducing a corresponding moment in an adjacent atom or ion, and thus lead to an attraction. In short, it is a combination of attractive and repulsive forces originating from the interpenetration of the electrical clouds of atoms that leads to the net attraction. With the exception of very polar molecules, and of hydrogen bonding, these forces account for 75-100% of the total cohesiveness of a substance. Electrostatic forces result from an electrostatic field that is superimposed upon that produced by dispersion forces in the case of an ionic solid material. If the molecule has a permanent dipole, the necessary charges are complete. If the molecule is not polar, the electrostatic field will induce a temporary dipole in it. The electrical field of force is equal to the force exerted on a unit-positive charge situated at the point in question. The value of the electrical field of force falls rapidly with distance, and even at a distance of only twice the shortest distance between ions, this force becomes negligible. Interaction of permanent dipoles leads to forces which depend upon the product of the strength of the two dipoles. Hydrogen bonding will take place if the negative pole of a strong dipole approaches the positive charged end of another dipole which consists of a hydrogen atom. The resultant force is a particularly strong interaction. This has its origin in the small size and mass of the hydrogen atom and the interaction is known as a hydrogen bond.

The mechanical interlocking of particles is the only bonding mechanism not involving atomic forces and is considered as being only a minor contributor to overall compact strength. In the pharmaceutical industry we must rely on this force to a great extent when we are making layered tablets or press-coated tablets. It is probably the mechanical interlocking of one layer to another or of the press-coat to the core that is responsible for the adhesion we observe. In the case of multi-layered tablets, bonding between the layers is optimum if the same material is used for each layer. Particles bonding through cold welding could only occur if the particles are the same material or if they have similar crystal lattices. In cases where the layer materials are different, optimum adhesion is obtained only if the first layer is incompletely compressed and can undergo further compression as succeeding layers are applied. The adhesion between layers is then probably caused by mechanical interlocking of the different materials. This is likewise true for press-coated tablets. The bonding of the press-coat to the core tablet would seem to be primarily dependent upon mechanical interlocking, with the exception of the fact that the press-coating is continuous around the core tablet, and does achieve a degree of integrity that is related to its own adhesive character. However, it is generally true that the amount of press-coating around the core tablet is rather minimal at the edges of the tablet, and the factor which seems to govern the overall behavior of the press-coated tablet is the adhesion of the press-coating to the core.

Dry adhesives or binders that are commonly used in pharmaceutical tabletting are materials such as microcrystalline cellulose, N.F., methylcellulose 400 cps, Solka-Floc, spray-dried lactose, pregelatinized starch, and many others. These ingredients have several properties in common that give them the ability to aid the compaction of tablets. They possess a low elastic modulus, and generally exhibit plastic deformation. If they are crystalline, they will either be symmetrically shaped such that in flowing they would acquire random orientation, thereby having random orientation of slip planes in the die cavity, or they would possess multi-directional slip planes within the individual crystal. These materials also exhibit high cohesive character within themselves, thereby strengthening the tablet matrix. Certain other substances aid compaction by acting other than binders or adhesives. These substances aid the movement of particles past one another during the initial stages of compression. Such substances as magnesium stearate, stearic acid or calcium stearate act in this manner. Generally, this beneficial aspect of their behavior is counterbalanced by the fact that they tend to decrease the cohesive character of the particle-to-particle bond by their presence between the particles. This effect was documented by Higuchi[8,9] in his series of articles on "The Physics of Tablet Compression." In order to tablet medicinal substances on high-speed tabletting equipment, it is often necessary to add excipients that improve flow and lubrication characteristics of the materials. It is quite often that these additives tend to inhibit compaction. These substances tend to function to decrease the cohesive or adhesive bond between particles of material being compacted (Figure 11). The materials themselves possess poor cohesive character and poor adhesive tendencies with other substances. materials of this type are substances such as the stearates, talc, starch, Cab-O-Sil, etc. The effect of magnesium stearate, which is a typical substance of this type, has been documented by

Shotton and Lewis (1964)[10]. They determined tablet strength of unlubricated materials under known compaction pressures and then compared the strength of these compacts with those prepared following lubrication with magnesium stearate. They reported that increasing the concentration of lubricant produced weaker tablets.

Initially, the powder bed, under an external compacting force, will transmit this force through interparticulate point contacts. Local deformation of the material will follow this concentration of stress at these points of contact. As discussed, this deformation will be either elastic, plastic, or destructive. The nature of the deformation will depend upon the magnitude of the force, the rate of application of the force, and the duration of the induced stress, as well as the physical properties of the material. As also previously stated, most materials do not exhibit pure responses and the real circumstance will probably be that it is a combination of these various types of deformation. The energy used in compaction will be expended primarily in overcoming the adhesive/cohesive forces between the particles as they change their relative packing positions, in deformation of the particles, and in fragmentation of the particles. It will also be expended in the subsequent aggregation and in overcoming elastic and residual stresses. This work was performed by Bal'Shin (1938)[11]. Other investigators concluded that the transmission of forces was also dependent upon the dimensions of the bed, the packing density, and the maximum particle size. Endersby[12] postulated that forces were transmitted in a zig-zag manner, mainly along the granular structures, and were resisted by the confining wall whenever the structure came into contact with it. Wulff

(1946 and 1949)[13,14] demonstrated the presence of radial and axial pressure gradients and was able to determine the stresses and strains in a compact from grid distortion measurements conducted using a deformable lead grid when filling the powder into the die cavity. Later, a measurement of the force transmitted through the powder bed to the bottom punch in a tablet machine was obtained both in the absence and presence of lubricants (Higuchi, 1954)[15]. Some preliminary measurements were also made of the die-wall pressure during compression, both with and without lubricants (Nelson, 1955)[8]. David Train, in this thesis work (1956)[1], set upon developing some reliable means of measuring the pressure at various points within a compact, and collecting experimental data to correlate these pressures to material properties. Train's work concluded that the applied pressure will be transmitted to the material immediately below the top punch, and subsequently passed on to the remainder of the powder bed. He also concluded that the radial stress has a specific relationship to the applied pressure, depending upon the physical properties of the material and the conditions of the surface of the punch and die walls. His most interesting conclusions (Figures 12 and 13) were obtained when he inspected the apparent densities within lubricated compacts, and concluded that in a compact there is a region of low density near the top center, and a region of high density about two thirds of the way down the compact. The greatest differences amounted to approximately 6%, and that if the regions of high density were correlated with more effective transmission of force, then the possibility exists for explaining phenomena such as "capping" and "laminating."

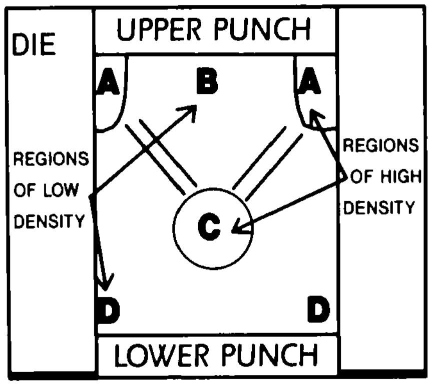

FIGURE 12

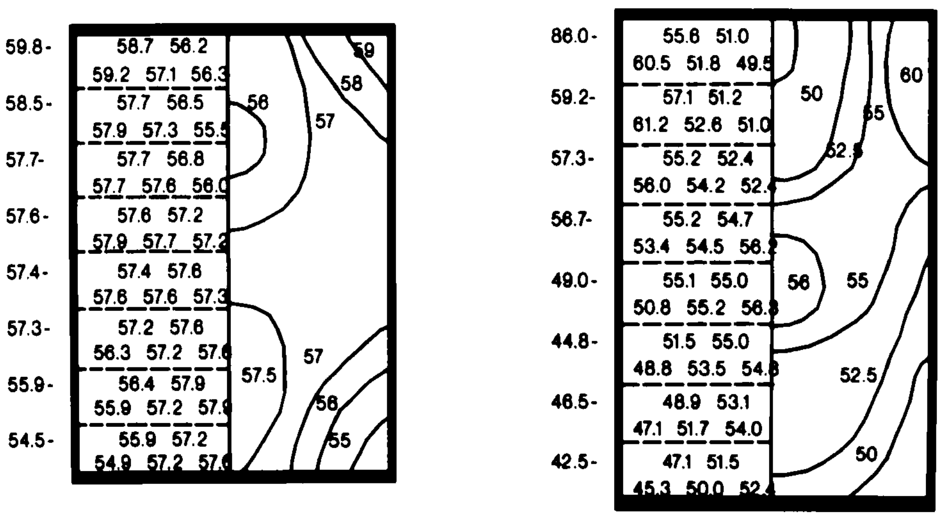

FIGURE 13

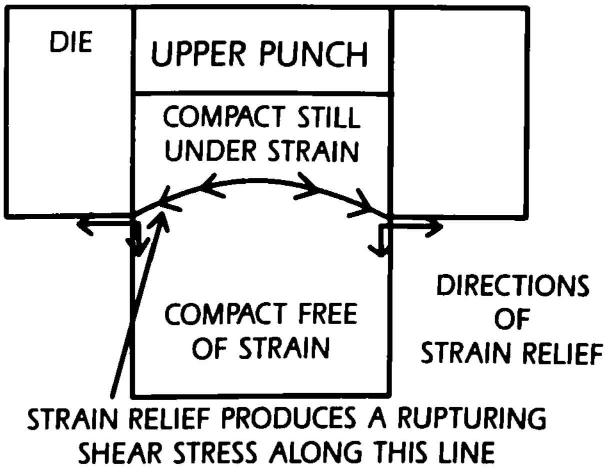

FIGURE 14

Special attention was given to the transmission of force in a compact in the direction normal to the direction of application. Long (1960)[16] advanced a theory to explain the die wall pressure during compaction in conventional dies. His work predicts that during the application of pressure and release of pressure by the punch, the radial pressure should follow a characteristic cycle, the form of which is determined by the deformation properties of the material (Figure 14). He also noted that at this stage the compact is no longer subject to compression from all sides. Under these conditions, friction between the die and the compact may have important defects. Long proposed that friction present at the die wall during ejection of the tablet could give rise to stresses within the compact which may be correlated with the capping phenomenon. This theory is somewhat in conflict with other capping theories. As mentioned previously, Train[1] revealed that there are appreciable density differences with the compacts compressed in dies,

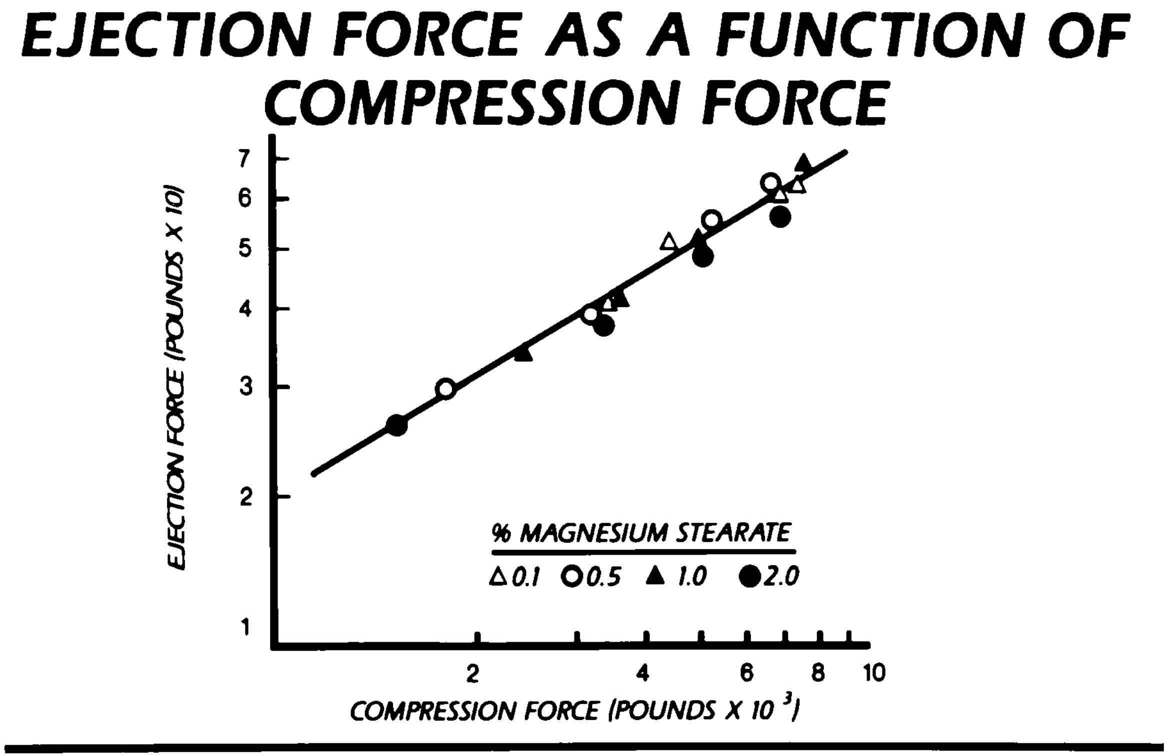
EJECTION FORCE AS A FUNCTION OF COMPRESSION FORCE
EJECTION FORCE (POUNDS X 10)
7
6
5
4
3
2
1
% MAGNESIUM STEARATE
Δ 0.1 O 0.5 ▲ 1.0 ● 2.0
2
4
6
8
10
COMPRESSION FORCE (POUNDS X 10 ³)

FIGURE 15

and that these density differences could be the explanation for capping that we observe periodically. Train's thesis work also proposed a theory which showed a linear relationship between the logarithm of ejection force, or force required to produce first movement, against the logarithm of compression force. This relationship has consistently been found to be true with instrumented rotary tablet machines (Figure 15).

It is well known that a powder mass undergoing compaction in a die exerts pressure on the die wall at right angles to the direction of compaction, and the die must be strong enough to withstand these radial pressures. It is also known that when the compression has been completed, and the punch removed from the die, the compact does not fall out of the die. The compact must be forced out, and the compact, as it leaves the die, expands. It is these facts which give rise to the problems in the pharmaceutical industry. Following the compression of the tablet in the die, it is necessary that the tablet be ejected from the die. To do this, the friction between the tablet and the die wall must be overcome, and the tablet must be able to withstand the expansion or elastic recovery which it will undergo following ejection. Consider the course of events as the compact is ejected from the die. As the compact exists in the die after the removal of the force from the upper punch, the relaxation of the compact begins. Obviously, it is initially only able to expand upward, and is restrained by the die wall and the lower punch on the other sides. The next event is the initiation of movement in the die, and we previously discussed it as the break-loose force or ejection force. the sliding of the compact upward in the die generates stress, and this stress radiates inward from the edges of the surface of the contact between the die and the compact. Then as the compact emerges from the die, the upper edge of this surface emerges from the die and a relaxation of the compact can progress in the radial directions as the tablet moves higher and higher in the die, until it is completely out of the die.

To review briefly, it is important to observe the compact relaxation characteristics at the point of peak compression, to observe the decay of radial die wall force after the removal of pressure from the upper punch, to measure the force required to initiate the ejection (break-loose force), to measure the sliding frictional forces required to eject the tablet from the die, and lastly, to characterize the strength of the compact after it is removed from the die. Many of these characteristics will be important to the characterization of the compaction characteristics of a material, and many of them are made use of in the presentations that will follow this overview today.

Next it is important to expand on the subject of the ejection process. The ejection process can be best characterized by describing it as a hammer blow to initiate the upward movement of the tablet in the die, followed by a sliding frictional phase which characterizes the compact and die wall properties, and thirdly, by a phase wherein the tablet first emerges from the die to the point that it is completely out of the die. The ideal wave form (Figure 16) which describes this event is shown here, and the various aspects of this wave form further delineate the terms that we have been using. The peak break loose force, as said previously, is the ejection force. The second segment represents the

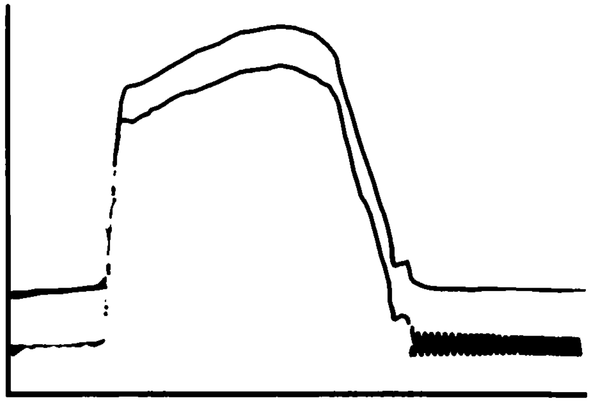

FIGURE 16

sliding frictional force, and you will note that the slope of this can be either a positive or a negative event, depending upon the material and the die wall condition. The third phase is a decay phase, wherein it ultimately goes to zero, and which signifies the tablet being completely pushed out of the die. The ejection event is the source of much stress in the compact, in that the compact is subjected to unequal distribution of force throughout the compact, and therefore unequal distribution of stress. It should also be noted that the sliding portion of the ejection event imparts tremendous shear to the very edges of the tablet, and this shear achieves consolidation that the compaction event is incapable of achieving. The ejection event is also associated with the generation of heat. This was studied extensively by Rankell and Higuchi (1968)[17], and they concluded that the mechanism of interparticulate bonding during tablet compression has not been satisfactorily explained, and although a number of theories have been advanced, the liquid surface cement theory is compatible with most of the known facts about tabletting. In addition, the possibility of formation of a liquid film at the particle surface is supported by thermodynamic analysis of the effects of stress distribution on melting point and solubility, and by heat transfer kinetics at the surface.

Ejection force is also a function of the lubrication properties of the material being compacted, and also the force transmission characteristics of the material. The lubrication of the material has two facets. Materials functioning as lubricants (magnesium stearate, etc.) will reduce the ejection force, but they also tend to reduce the interparticulate cohesion characteristics, thereby reducing the compact strength. These same materials also can facilitate the transmission of force through the compact by making it easier for particles to move within the confines of the compact prior to particle-to-particle adhesion being established.

Previously in this paper the magnitude of the force applied and the rate of the application of the force were discussed. A typical force time wave form is shown in Figure 6. Both of these factors are machine dependent. Various tabletting machines have different abilities with regard to the magnitude of compaction force and the rate at which the force is applied on that machine. The other aspect is that to compensate for the inability of a machine to generate the amount of force required or for the machine to apply the force at an optimum rate, equipment manufacturers have added what is termed "precompression" capability to machines. This accomplishes, in a mechanical way, what in theory is required to achieve the desired tablet characteristics when, in its absence, the machine would be incapable of compressing the material adequately. In essence, the precompression capability enables the machine to increase the amount of work performed on the compact. It increases the amount of time the material in the die is under pressure. This means that the adhesion and cohesion properties of the compact are improved over what would be achieved if precompression was not utilized. The other way equipment manufacturers assist in manufacturing tablets with the desired characteristics is by modifying the way the force is applied. The most recent innovation is to activate the punches via hydraulic means, so as to generate a compression force wave form that is essentially a square wave rather than a sine wave, and thereby maximize the amount of work done on the compact in a given amount of time. Other machine manufacturers have modified compression roller-type mechanisms to increase the work function by going to ever larger diameter rollers and to, as mentioned previously, precompression systems. Equipment characteristics can also affect ejection of the tablet from the die. This is a rate phenomenon also, and the angle of the ejection cam is a definite factor in the ejection characteristics of the material. This would also be dramatically affected by the machine head speed, since the rate at which the punch passes up the ejection cam would also govern the rate of movement of the tablet in the die. Modern machines also have the provision for controlling the depth of compression in the die, and this attribute can minimize the ejection path length, but cannot compensate for the rate of ejection.

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